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MICROBIOLOGICAL METHOD FOR PRODUCTION OF OIL HYDROCARBONS AND SEPARATE
HYDROCARBON FRACTIONS FROM SOLID FOSSIL FUELS
[MIKROBIOLOGICHESKIY SPOSOB POLUCHENIYA UGLEVODORODOV NEFTI I
OTDEL'NYKH UGLEVODORODNYKH FRAKTSIY IZ TVERDYKH GORYUCHIKH
ISKOPAYEMYKH]

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The invention concerns methods of processing solid combustible fossils by means of microorganisms and solves the task of producing hydrocarbons identical to those contained in petroleum of natural origin and individual fractions thereof.

The capacity of microorganisms for intracellular synthesis of hydrocarbons (E.G. Dedyukhina, V.K. Yeroshin. Biosintez uglevodorodov mikroorganizmami (Biosynthesis of hydrocarbons by microorganisms). Uspekhi sovremennoy biologii AN SSSR, 1973, vol. 76, no. 3(6), p. 351-361).

A method of microbiological synthesis of oil out of materials of animal origin by treating them at high temperature and pressure ($P=3-30$ atmospheres; $t=100-350^{\circ}\text{C}$) in the presence of plant residues (DE2455368, application published 1976).

This method is complicated and unsafe, since it requires the use of super-high temperatures and pressure. A hydrocarbon mixture close in composition to petroleum is obtained.

The impossibility of directly controlling the process of producing hydrocarbons is a disadvantage of the method.

A microbiological method of acting on solid fossil fuels with cultures of the bacteria *Thiobacillus aquaesulis*, or *Thiobacillus denitrificans*, or *Thiosphaera pantropha* in any combinations is proposed for producing a wide range of petroleum hydrocarbons of petroleum and individual fractions thereof.

The method consists in treating suspensions of ground raw material (solid fossil fuels) with cultures of *Thiobacillus aquaesulis*, or *Thiobacillus denitrificans*, or *Thiosphaera pantropha*, taken in any combinations, in the presence of compounds that are

hydrogen donors.

The process is conducted at temperatures of 4-60°C (temperature optimum 18-24°C) and at a pH of 6-9.

The following are used as hydrogen donor substances: acids, phenols, naphthalene, tetrahydronaphthalene, cresol.

Solid fossil fuels - humites, anthracite and bituminous coal, peat - are used as the raw material for producing petroleum hydrocarbons.

Bacteria of the species *Thiobacillus thioparus* also are used additionally in the case of using peat as a raw material.

The method is performed in the following way.

Solid fossil fuels are subjected to mechanical crushing, then liquid - water, inorganic (sulfuric, nitric, etc.) acid, or any liquid hydrocarbon - petroleum or any petroleum fraction, is added in order to create a suspension or paste. Weak or more concentrated acids are used. The acids are used as diluting agents (as the liquid phase) and as hydrogen proton donors. Before mixing them with solid fuel in the liquid phase of the acid, a culture of *Thiobacillus aquaesulis* bacteria is added to liquid hydrogen or in petroleum individually or in combinations with bacteria of the species *Thiosphaera pantophora*. Then the liquid and solid phases are mixed once by means of using a mixer, an air pump (barbotation), or gas pump - decanting the suspension, or in another way. The bacterial cultures used are preadapted to hydrocarbon and organic (coal-peat) media.

According to a second version of the method, a bacterial culture is added to a prepared aqueous-organic (water-coal, water-peat, etc.) medium (suspension), to an acid-organic, or to a hydrocarbon-coal

(peat etc.) medium. Previously cultured cultures of bacteria in water, an aqueous solution of acid, or hydrocarbon (petroleum, etc.) according to the first version of the method is technically easier and improves the mass-exchange of the process and therefore is preferably.

The solid fossil fuels are mixed with liquid in order to provide contact of the bacterial culture dissolved in the liquid phase with maximum amount of surface of the solid substrate of the raw material (coal etc.) and in order to provide maximum contact of the bacteria with the macromolecules and molecules of solid fossil fuels.

Organic or inorganic acid in an amount of 1-2% of concentrated 96% acid is added to solid fossil fuel organic material as a hydrogen donor in the system, for example, an aqueous 1-2% solution of acid is added to the solid fossil fuel material in a ratio of 1:1-1.5 (0.75-1 liter of solution per 1 kg of solid fuel). In the case of using water or weak acid solutions as the liquid medium, on the whole the water-organic (coal, peat, etc.) suspension is converted into a water-hydrocarbon emulsion. This emulsion contains water (a mixture of industrial water - diluting agent and water in the form of moisture initially included in the composition of certain high-moisture solid fossil fuels), molecules of individual hydrocarbons, and extensive petroleum fractions, obtained from solid fuel raw material under the action of the above-mentioned bacteria, and also a slurry consisting of coal ash (metals, sulfur, oxidized compounds). Then hydrocarbons (synthetic petroleum) are separated from the water by any traditional industrial methods - a settling period, use of deemulsifiers, separation, etc. It is preferable to use petroleum or any liquid hydrocarbon substances that are added to the solid fossil raw material

in different economic proportions (from 30 to 100% petroleum to the solid fossil fuel material), sufficient for creating a paste (suspension), as the liquid medium for bacteria in order to eliminate the process of dehydration of the synthetic petroleum obtained.

As a result, a homogeneous hydrocarbon material - synthetic petroleum (the set of fractions and individual hydrocarbon substances), the components of which are hydrocarbons and petroleum fractions, obtained under the action of bacteria from solid fuel raw material, and petroleum or a petroleum product, uses as a diluting agent (liquid phase) are obtained as a result. Hydrocarbons obtained from coals are organically mixed with the initially poured petroleum (petroleum product), mutually dissolving, in this case the total volume of the petroleum (petroleum product) is increased correspondingly. The petroleum or petroleum product used at the beginning of the process as a diluting agent (liquid phase), either is removed without mass losses from the synthetic petroleum obtained, or is used together with the latter as a single petroleum raw material (for distillation, chemical processing, heating, and other purposes).

The volumetric ratio of gasoline and diesel fractions included in the petroleum - diluting agent remains unchanged in the course of the entire process.

The amount of benzene, its uninucleate homologues, and isoparaffin compounds is increased in relation to the normal paraffin and naphthalene hydrocarbons in the gasoline fraction of petroleum or any other petroleum product, among them in the pure gasoline fraction, used as a diluting agent (liquid phase), as a result of performing the method, under the action of bacteria of the species *Thiobacillus*

aquaesulis. Increasing the amount of the lower (uninucleate) aromatic compound and isoparaffin hydrocarbons in the gasoline fraction leads to an increase in its octane number and fuel grade. The benzene homologues and isoparaffin substances formed are a more valuable raw material than the original compounds: condensed cycles and normal paraffin compounds.

The process time for forming synthetic petroleum and petroleum products depends on the degree of pulverization of the solid fossil fuels, the seeded bacteria concentration, the material exchange indicators (which depend both on the initial and on the subsequent mixing), and the presence of a hydrogen source in the system (hydrogen donor). Optimal parameters are: size of the pieces of solid fossil fuel no more than several cubic millimeters, seeded bacteria concentration not less than 10^4 per ml of medium, or 1 kg of dry preparation (several billion bacteria in 1 ml), good initial and subsequent constant mixing (or pure periodic mixing), hydrogen donor concentration in mass volume not less than 1-2% (optimum 4-7%) in the organic mass of the solid fossil fuel (for example, 4-7% solutions of organic or inorganic acids in a volume 1:1 to the mass of coal, or phenol, cresol, naphthalene, or tetrahydronaphthalene in an amount of no less than 1-2 kg per 100 kg of organic material of solid fossil fuel). In the case of the given optimal ("ideal") conditions the process of forming petroleum hydrocarbons from solid fossil fuels takes place in the reaction zone in 6-24 hours. The attainable yield is 90% of the weight of raw material.

The active mechanism of the bacteria is to break down the macromolecules (molecules) of solid fossil fuels which, according to

the traditional data, are very condensed polycyclic and polyaromatic systems. The bacteria break down the carbon-carbon (C-C) bonds and the heteroatomic bonds of solid fossil molecules. As a result less condensed systems of lower molecular weight takes place up to different uninucleate aromatic compounds, petroleum and liquid paraffin hydrocarbons, forming different petroleum fractions. Removing sulfur, nitrogen, and oxygen atoms from the condensed systems, the bacteria break heteroatomic bonds, which leads to breakdown of the condensed systems and reduction of the molecular weight of the latter.

By breaking the oxygen, sulfur, and carbon-carbon bonds and transferring the electrons in the organic material, the bacterial strains used contribute to the formation of free radicals, according to which hydrogen from an external source-hydrogen donor is added (stabilization of radicals with hydrogen). Hydrogenation of solid fossil fuels takes place. In a system with the given species of bacteria the hydrogen donor "transfers" to the solid fossil fuel 5-10% (by weight) of the hydrogen (hydrogen content of the organic material increases by 5-10%). As a result the organic material of the solid fuels is converted into hydrocarbons, in particular into liquid hydrocarbons.

The process of forming low-boiling hydrocarbons, synthetic petroleum (liquid hydrocarbon raw material) and its derivatives from solid fossil fuels, in particular light fractions is performed in three versions, which may be observed and fixed on the super-molecular and visual levels:

1. direct formation of liquid petroleum hydrocarbons of petroleum

(on the chemical molecular level it is possible via the stage of intermediate products) in the form of low-boiling fractions, or in the form of a wide range of fractions - synthetic petroleum, with a yield of 50-90%, calculated according to the dry weight of the coal (the process is performed with peat or brown coal in anaerobic conditions, in aerobic conditions with anthracite coal), in both cases in the presence of a hydrogen donor the process time is reduced two-fold.

2. formation of fuel oil as an intermediate product (process time 2-4 weeks in anaerobic or aerobic conditions) which then in the course of performing the method is subjected to bacterial decomposition of the hydrocarbon molecules (biocracking) and bacterial hydration (biocatalysis) with the formation of more liquid and low-boiling hydrocarbons and petroleum fractions (2-4 more weeks in anaerobic conditions with donor hydrogen);

3. formation of fuel oil, as an end product, with up to a 80-95% yield of the latter as compared with the initial solid raw material - dry weight of coal or other solid fossil fuels (process time 2-4 weeks in anaerobic conditions, or in aerobic conditions, with donor hydrogen or without it).

4. formation of tar (anthracite pitch) analogs with a 90-95% yield as compared with the initial raw material. This intermediate material contains both uninucleate and multinucleate aromatic and petroleum cycles, both liquid and solid paraffin hydrocarbons, phenols, pitches, and neutral substances. The tar obtained according to the given method visually represents a fairly uniform viscous liquid material, as opposed to the precursor already pertaining to the hydrocarbon raw material. The tar can be final products (because of

the artificial stopping of the process), or is further subjected to bacterial decomposition and hydration, with the formation of individual hydrocarbons, included in gasoline, diesel, and fuel oil fractions (in a wide fraction of synthetic petroleum).

In the case where the process is stopped in the tar stage (for example, by limiting the process time - less than 1-2 weeks), the product obtained can serve as fuel or raw material for traditional processes of hydration - destructive hydrogenation (obtaining liquid hydrocarbon fuels and individual substances). It is desirable for liquefaction of the tar to take place by prolonging the process with the addition of a hydrogen donor (in the case of an initial absence thereof). Further destruction of large molecules, hydration and lightening of the molecular hydrocarbon composition with the formation of petroleum fractions takes place. This bacterial process removes heteroatoms - sulfur, nitrogen, oxygen, from the tar molecules, decomposition of pitches and high molecular weight compounds, and conversion of tar into liquid petroleum fractions.

Experiment. Bacteria of the species *Thiobacillus aquaesulis* and *Thiosphaera pantotropha* were adapted to hydrocarbon and coal-peat media, as a result the strains *Thiobacillus aquaesulis*-39' and *Thiosphaera pantotropha*-66, capable of existing in the given media, and utilizing molecules of solid fossil fuels, bitumens and petroleum in their vital activity, were obtained.

In coal-petroleum and water-coal suspensions a culture of *Thiobacillus aquaesulis* bacterial was added separately or in combination with a culture of bacteria of the species *Thiosphaera pantotropha* in the form: pure separated culture; together with a

liquid nutrient medium. The bacterial concentration was taken as being no lower than 10^4 bacteria per 1 ml of medium, or 1 kg of separated biomass per 10-60 tons of bituminous or anthracite coal, peat, or any other solid fuel substrates.

A gradual transformation of the molecules of solid fossil fuels (coal, peat, etc.) into hydrocarbon molecules and the formation of hydrocarbons containing synthetic petroleum takes place in the course of 1-2 months without mixing the system.

In the case of an initial introduction of a hydrogen donor substance into the system in an amount of 1-5% of the weight of solid fossil fuel and with constant or partial periodic mixing (5 minutes every 2-4 hours), conversion of solid fossil fuels into hydrocarbons began after 7 hours, the formation of the hydrocarbon material completely stopped after 7 days.

Examples

The following forms of raw material were taken for the experiment:

peat of small local deposits in Tataria and peat from commercial depositions in Byelorussia, Ryazan, Tyumen, and Novgorod provinces.

Test sample of peat. Elementary composition, % of original peat material (transitional type of peat, that is, between high and low): C 58.6%, H 6%, O 32.8%, S 0.4%, N 2.2%. True density 1.5. Combustion heat 7.5 MJ/kg.

Bituminous coal from the Moscow coal basin (B2 industrial group), density 1.5 g/cm^3 , combustion heat 10.9 MJ/kg. Elementary composition of test sample, % of coal fuel weight: C 75%, H 5.5 %, O 18.5%, N 1.9%.

Bituminous coal from the Kansk-Achinskii coal basin (B1 and B2), density 1.6 g/cm³, combustion heat 11.8-15.6 MJ/kg. Elementary composition of test sample, % of coal fuel weight: C 75%, H 5.5 %, O 18.5%, N 1.0%. Combustion heat of test sample 6600 kcal/kg.

Bituminous coal from Orenburg province, B1 and B2 (Tyul'ganskiy coal section), density 1.8 g/cm³, moisture up to 54%, ash content 24-30%, combustion heat 1850-1900 kcal/kg.

Anthracite coal from the Kuznetsk coal basin, grade D (long flame), G (gas), Zh (liquid), KZh (coke-fatty), K (coke), T (lean).

Elementary composition, % of coal fuel weight:

grade D: C 80%, H 5.5%; O+S org. = 12.7%, N 1.8%. Combustion heat 7950 kcal/kg.

Grade G: C 84%, H 5.0%, O + S 9.3%, N 1.7%. Combustion heat 8200 kcal/kg.

Grade Zh: C 87%, H 5.0%, O+S 6.3%, N 1.5%. Combustion heat 8450 kcal/kg.

Grade KZh: C 87.5%, H 4.9%, O+S 6.1%, N 1.5%. Combustion heat 8500 kcal/kg.

Grade K: C 88%, H 4.8%, O +S 6.0%, N 1.5%. Combustion heat 8600 kcal/kg.

Grade T: C 91%, H 4.0%, O+S 4.0%, N 1.0%. Combustion heat 8500 kcal/kg.

Anthracite coal from the Donetsk coal basin, combustion heat 21.2-26.1 MJ/kg:

Grade D-T and A (anthracite) grade D (long-flame), G (gaseous), Zh (fatty), K (coke), OS (lean caking), T (lean), and A (anthracite).

Elementary composition, % of coal fuel weight:

Grade D: C 80%, H 5.5%, O+S org. 12.7%, N 1.8%. Combustion heat 7950 kcal/kg.

Grade G: C 85%, H 5.0 %, O+S 9.3%, N 1.7%. Combustion heat: 8200 kcal/kg.

Grade Zh: C 87%, H 5.0%, O+S 6.3%, N 1.5%. Combustion heat 8450 kcal/kg.

Grade K: C 88%, H 4.8%, O+S 6.0%, N 1.5%. Combustion heat 8600 kcal/kg;

Grade OS: C 89%, H 4.5%, O+S 5.0%, N 1.5%. Combustion heat 8600 kcal/kg.

Grade T: C 91%, H 4.0%, O+S 4.0%, N 1.0%. Combustion heat 8500 kcal/kg.

Grade A: C 93%, H 3.0%, O+S 3.0%, N 1.0%. Combustion heat 8200 kcal/kg.

The wide range of hydrocarbons obtained (synthetic petroleum) and fuel oil independent of the type of solid fossil fuel practically did not differ from one another according to physical-chemical characteristics, but depending on the type of raw material differed from one another according to the group hydrocarbon composition (that is, according to the aromatic, methane, and petroleum hydrocarbon content). Yield: 1 t of the organic part of solid fossil fuels - 90% hydrocarbons, 1 t of coal + 1 t of 2% acetic acid + 100 g of biomass, yield 800 kg of a wide range of hydrocarbons, 1 t of peat + 1 t of 2% acetic acid + 100 g of bacterial biomass, yield 200 kg of a wide hydrocarbon fraction.

The physical-chemical characteristics of the synthetic petroleum obtained, that is, the entire set of fractions obtained and individual

hydrocarbons (initial material - peat or bituminous coal, or anthracite coal, or fuel shale).

Chromatography of the gasoline fraction

Hydrocarbon content 84%, hydrogen content 14%, sulfur content 1.5%, nitrogen and oxygen content 0.5%.

Density - 0.85-0.95

Viscosity - 5-15 cSt (50°C)

sulfur not more than 1.8% - (on the average 1-1.5%)

resins - 8-15%

asphaltenes - 0.5-1.5%

cokeability - 3-8%

combustion heat - 10400-11000 kcal/kg

Yield of fractions, weight % n.k. 28-200°C=22%, 28-300°C=38%, 28-350°C=50%, above 350°C=50%.

Otherwise (the same): 28-200°C=22%, 200-300°C=16%, 300-350°C=12%, above 350°C=50%.

Differences in the group hydrocarbon composition:

The synthetic petroleum obtained from peats: is characterized by the following group hydrocarbon composition (total content of substances of different classes in all fractions):

Aromatic hydrocarbons 45%, methane 30%, petroleum 25%. Solid paraffin: 3%.

Synthetic petroleum obtained from bituminous coals: is characterized by the following group hydrocarbon composition (total content of substances of different classes in all fractions):

Aromatic hydrocarbons 30%, methane 50%, petroleum 20%. Solid paraffins: 5%.

Synthetic petroleum obtained from hard coals and anthracites is characterized by the following group hydrocarbon composition (total content of material of different classes in all fractions):

Aromatic hydrocarbons 50%, methane 20%, naphthene 30%. Solid paraffins: 2%.

Physical-chemical characteristics of the synthetic fuel oil obtained.

A fuel oil analogous to M-100 fuel oil is obtained: yield: 90% of the organic raw material.

Carbon content 86%, hydrogen 12%, sulfur 1.5%, nitrogen and oxygen no more than 0.5%.

Density at 20°C - 0.950-0.990

Viscosity of the VU condition - 15.5 (80°C)

Ash content - 0.30-1.0%

Sulfur no more than 2% - (on the average 1-1.5%)

hardening temperature- +25°C

flash temperature - +125°C

Combustion heat - 9500 kcal/kg

pitch - 12-15%

asphaltenes - 0.5-1.5%

carbenes and carboids - 1.3%

excise pitches - 28%

coke - 10-15%

The synthetic fuel oil obtained from peats is characterized by the following group hydrocarbon composition:

Aromatic hydrocarbons 45%, methane-naphthene hydrocarbons 55%.

The synthetic fuel oil, obtained from brown coals, is

characterized by the following group hydrocarbon composition.

Aromatic hydrocarbons 40%, methane-naphthene hydrocarbons 60%.

Synthetic fuel oil obtained from hard coals and anthracites is characterized by the following group hydrocarbon composition:

Aromatic hydrocarbons 60%, methane-naphthene hydrocarbons 40%.

Physical-chemical characteristics of the tars obtained:

Yield: 90% of the organic weight of raw material

- peat tar:

density - 0.95-1.05 g/cm³

viscosity - 10 cSt (50°C)

sulfur - 9.5%

combustion heat - 8700-9000 kcal/kg.

Hydrocarbons around 40% including aromatic, saturated and unsaturated:

phenol - 20%

solid paraffin - 3.0-8.0%

Oxygen compounds around 40% including:

acids and phenols - 10%

asphaltenes 0-2% - brown coal tar:

density - 0.9-1.00 g/cm³

viscosity - 5 cSt (50°C)

sulfur 0.5%

combustion heat - 9900-10000 kcal/kg.

Hydrocarbons around 40%, among them aromatic, saturated and unsaturated:

phenols - 8%

solid paraffin - 7.0-18%

oxygen compounds around 16%, including:

acids and phenols - 10%

asphaltenes - up to 5% - hard coal tar:

density - 0.96-1.08 g/cm³

viscosity - 15 cSt (50°C)

sulfur 1.5%

combustion heat - 9400 kcal/kg.

Hydrocarbons around 40%, among them aromatic, saturated, and unsaturated

phenols - 15%

solid paraffin - 4-6%

Oxygen compounds around 30%, including:

acids and phenols - 10%

asphaltenes up to 30% - shale tar:

density - 0.95-1.00 g/cm³

viscosity - 10 cSt (50°C)

sulfur 0.9%

combustion heat - 9800 kcal/kg.

Hydrocarbons around 40%, among them aromatic, saturated and unsaturated

phenols - 15%

solid paraffin - 0-1%

Oxygen compounds around 30%, including:

phenols - 15%

solid paraffin - 0.1%

oxygen compounds around 30%, including:

acids and phenols - 10%

asphaltenes up to 15%

Determination method:

The carbon and hydrogen content of coals and in the hydrocarbons obtained (in synthetic petroleum, in fuel oil and in tar) was determined by the spectral method and the method of burning a weighed amount in an oxygen flow - "pyrolytic burning".

The sulfur content in the petroleum investigated was determined by the method of burning in a quartz tube, in fuel oil and tar - burning in a tube oven (GOST 1437-56) and burning in a calorimetric bomb (GOST 3877-49). The mercaptan and hydrogen sulfide content of the petroleum was determined by the potentiometric method. The nitrogen content of the petroleum was determined by the Dumas method.

The pitch-asphalt substance content was determined by the adsorption method (adsorption on silica gel). The composition of the pitch-asphalt substances was determined by Markussan analysis. The asphalt content was determined by the Gold "cold method". The density of the artificial petroleum was determined by the pycnometric method on a pycnometer, the viscosity - by the method of determining the conditional viscosity, with a VU type viscometer. The molecular weight of the pitches was determined on a mass spectrometer and by the cryoscopic method. The density of the pitches was determined by the pycnometric method. The sulfur content of the pitch-asphalt substances was determined by the method of burning in a quartz tube, the nitrogen content by the Dumas method, the oxygen content was determined (calculated) according to the element difference, and also was determined in analyzers of the magnetic and polarization type.

Formulation of Invention:

1. A microbiological method of producing petroleum hydrocarbons and individual hydrocarbon fractions of solid fossil fuels, wherein a suspension of solid fossil fuels subjected to grinding is treated with cultures of *Thiobacillus aquaesulis*, or *Thiobacillus denitrificans*, or *Thiosphaera pantotropha* bacteria, taken in any combinations, in the presence of a compound that is a hydrogen donor.

2. The method according to paragraph 1, wherein the process is conducted at 4-60°C preferably at 18-24°C, and at pH 6-9.

3. The method according to paragraph 1, wherein humites, hard and brown coals, and beat are used as the raw material

4. The method according to paragraph 1, wherein acids, phenols, naphthalene, tetrahydronaphthalene, and cresol are used as hydrogen donor compounds.

5. The method according to paragraphs 1-4, wherein peat is used as the raw material, and the above-mentioned cultures of bacteria are used together with bacteria of the species *Thiobacillus thioparus*.